Tracking the Origin of Crude Samples

by

Nordiana binti Mohamad Yusof

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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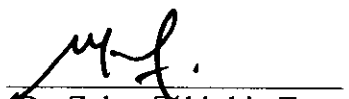
Approved by,



(Dr. Zuhar Zahir bin Tuan Harith)

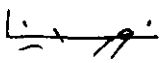
UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

January 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.



NORDIANA BINTI MOHAMAD YUSOF

ABSRACT

The report describes briefly the background of the project which mentions the definition of oil seep and Labuan geological setting. The project is about studying the relationship between the three given crude samples with the samples from the three oil fields near Labuan and other five oil fields. The report also informs about the project's problem statement, objectives and scope of study of the research. Under the scope of study, the report explains the relevance of the project and its feasibility within the time frame of ten months. The favoured chemical analysis is discussed in the literature review. The project activities, tools required and key milestones are stated in methodology part of the report. It is concluded that the unknown samples A, B and C are from Angsi, Baronia and Dulang oil fields respectively.

ACKNOWLEDGEMENTS

I would like to express my deepest appreciation to my supervisor Dr. Zuhar Zahir bin Tuan Harith for continually convincing me to pursue this project till the finish line. Without his guidance and persistent help, this dissertation would not have been possible.

I would like to thank Dr. Awang Sapawi and Mr. Salleh Bador of PETRONAS Research and Services Sdn. Bhd. (PRSB) for paying the most attention at me during my visit there to inquire more about gas chromatography and for producing to me GC results of the available crude samples.

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INTRODUCTION

1.1 BACKGROUND OF STUDY

1.1.1 Definition of Oil Seep

Generally, an oil seep is a site on any geologic structure where liquid hydrocarbons escape to the surface through fractures and fissures in between geological structures. Oil seep is very common and much of the petroleum discoveries during the 19th century were from observations of seeps. Oil seeps may contribute to pollution significantly especially if the seeps are offshore and can be mistaken with oil spill from tankers and oil platforms.

Answers.com defines oil seep as *"The emergence of liquid petroleum at the land surface as a result of slow migration from its buried source through minute pores or fissure networks"*.

Clarke & Cleverly (1990) meanwhile defines seep as *"the surface expression of a migration pathway, along which petroleum is currently flowing, driven by buoyancy from a sub-surface origin"*.

Natural Oil and Gas Seeps in California on the other hand defines seeps as *"... natural springs where liquid and gaseous hydrocarbon leak out of the ground. Whereas freshwater springs are fed by underground pools of water, oil and gas seeps are fed by natural underground accumulations of oil and natural gas"*.

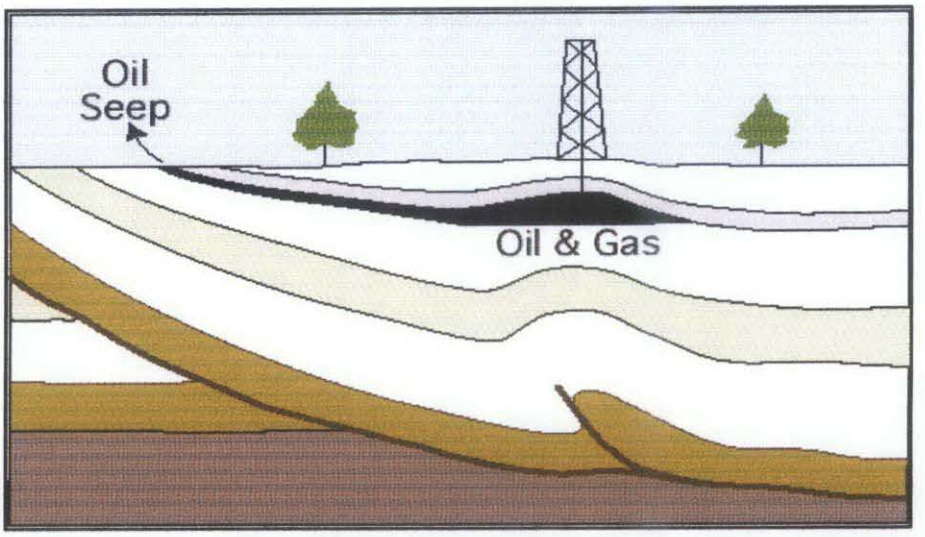

Figure 1.1: A vertical slice through the Earth's crust showing folded layers of sedimentary rocks holding oil and gas in the crest of an underground fold.

There are times when the oil that leaks to the Earth's surface is finally transformed from a clear fluid to a tar-like substance called asphaltum. The lighter components of the oil are lost to evaporation, and the remaining heavier oil is oxidized and degraded by bacteria until it becomes sticky and black.

Oil seep can also occur offshore. Reddy, C. (2009) explains that *"...this oil spill was entirely natural. The oil had seeped from reservoirs below the seafloor, leaked through cracks in the crust about 150 feet (45 meters) under water. Lighter than seawater, the escaped oil floated to the ocean surface"*. Figure 1.2 shows the oil leaving the sub seafloor travels through the water column to the surface and ultimately sinks in a plume shape onto the seafloor where it remains in the sediment.

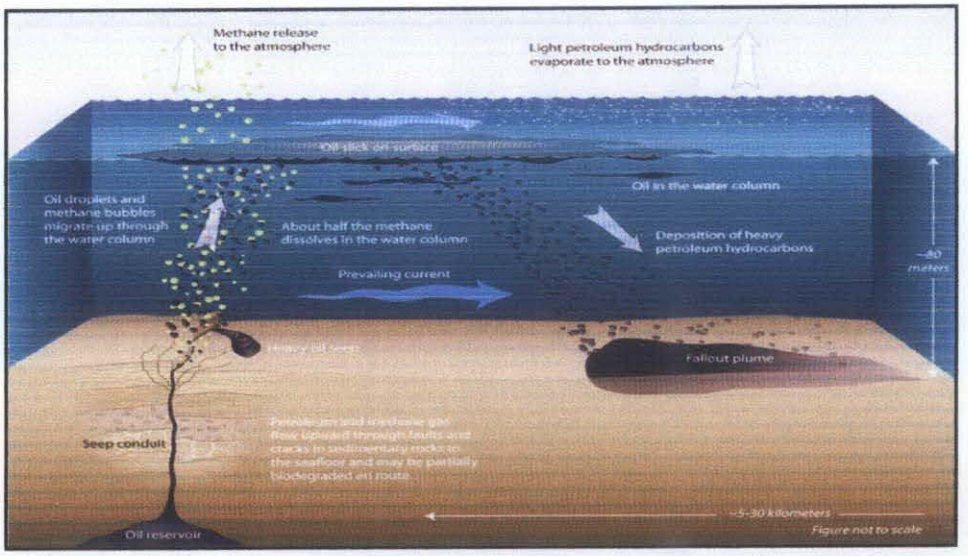

Figure 1.2: The route travelled by oil leaving the sub seafloor reservoir.

The knowledge that surface seepage has a direct link to subsurface oil and gas accumulations is not new and has been the stimulus for many of world's early major explorations by the industry's pioneers as far back as the 1860's in Pennsylvania and Azerbaijan (NPA Satellite Mapping).

The following decades spanning the late 19th century and the first half of 20th century heralded the first discoveries. Surface seepage was associated with Sumatra (1885), Texas (Spindeltop, 1901), Oklahoma (1905), Persia (Majid-I-Sulaiman, 1908), Mexico (Golden Lane, 1910), Venezuela (Los Barros, 1922), Iraq (Kirkuk, 1927), Bahrain (1932) and Kuwait Burgan, 1938).

NPA Satellite Mapping states that the work published by BP and others in the early 90's demonstrated that over 75% of the world's petroliferous basins contain surface seeps, the exceptions being those with unbroken regional evaporitic seals (such as the Hith Anhydrite in the Arabian-Iranian Foreland basin). Most seeps represent tiny but detectable volumes of oil and gas which are not significantly depleting the reservoir.

Exceptions would be in some recent onshore fold and thrust belts where accumulations have either been breached or redistributed to tertiary traps and where the link between surface seeps and the leaking traps is more complex. Such geology, however, is rarely encountered in offshore basins so that problem does not arise. Confirmation of the presence of seeps, therefore especially in offshore basins, is

positive and in the vast majority of cases is not indicative of breached or depleted traps.

1.1.2 Labuan Geological Setting

The island of Labuan is the second Federal Territory of Malaysia located 10 km off the coast of Sabah and covers an area of 98 km^2. Labuan settles on Sabah basin where its deltaic sediments produce some oil from sandstones. Seven oil fields are channelled into Labuan Terminal.

Labuan is essentially a northeastward-plunging, asymmetrical anticline and is one of the many compressional anticlines formed within the Middle Miocene-Recent succession of the Sabah basin (Bol & Van Hoorn, 1980). The core of Labuan anticline is formed by the Upper Oligocene-Lower Miocene Temburong Formation. Hence, the topography east of Labuan City is rather subdued because of the shaly lithology of the underlying rocks. Labuan is composed of alternating beds of clay and sandstones.

The two limbs of the Labuan anticline are formed of the more sandy and conglomeratic Temburong and Belait formations. These formations are well exposed in beach cliffs along the shores of Tg. Punai, Tg. Layang-Layangan and Tg. Kubong. Generally the Temburong formation is more strongly deformed, indurated and often steeply dipping while the Belait formation has gentler dips implying that they were subjected to different degrees of deformation.

Major hydrocarbon accumulations in the NW Sabah basin have been produced from reservoirs range from coastal/shallow-marine sediments to deep marine turbidites. The major fields near the island are Semarang, Kinabalu and Sumandak fields which sit on East Baram Delta. Meanwhile, Kikeh and Gamusut-1 are the two newly discovered deepwater fields found by Shell and Murphy Oil respectively.

There is appearance of oil seeps in Labuan. Labuan water accommodates a few oil blocks. Thus, these oil seeps might originate from the surrounding oil fields.

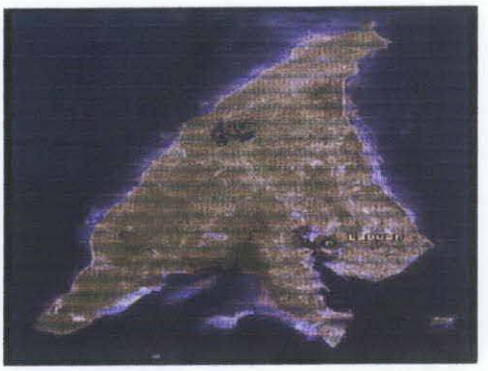

Figure 1.3: Labuan Island.

Figure 1.4: The oil seep site in Labuan.

Figure 1.5: The oil seep on site.

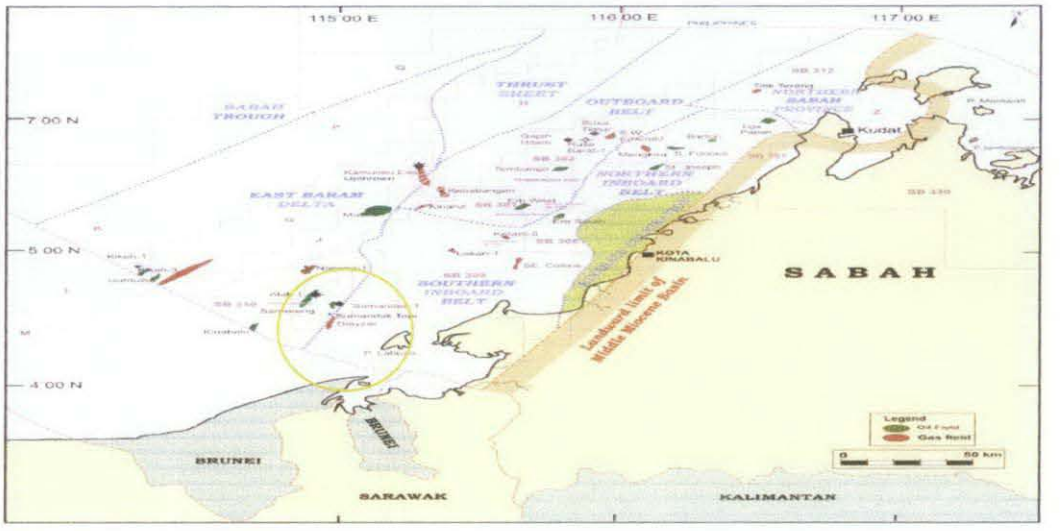

Figure 1.6: The oil fields surrounding Labuan which are Sumandak, Semarang and Kinabalu.

1.2 PROBLEM STATEMENT

There are three crude samples of unknown origin. It is unsure if they come from oil seep in Labuan. Therefore, chemical analysis is conducted unto all the possible candidates of crude oil. There are three nearest fields to Labuan which are Kinabalu, Semarang and Sumandak with other five probable oil fields which are Angsi, Baronia, Bokor, Dulang and Tapis. Are any of these fields related to the three unknown crude samples?

1.3 OBJECTIVES AND SCOPE OF STUDY

There are two paramount objectives that make up the foundation of this research project. They are as follow:

1) To perform chemical analysis on the unknown crude samples and the samples of oil taken from three oil fields near Labuan and other five probable oilfields.
2) To determine the correlations between the unknown crude samples and the eight oil fields crude samples.

The scope of study for this project involves reviewing previous literature on various chemical analyses done to trace oil source. In addition, oil samples data from the eight oil fields are retrieved. Then, laboratory test is performed unto all the unknown samples. The eight samples from the eight fields are compared to the unknown samples. Report writing is done throughout progression and completion of the project. This project is expected to prove whether the three unknown samples originate from any of the eight fields.

CHAPTER 2
LITERATURE REVIEW

2.1 FRACTIONATION METHOD

The components of mixture can be separated into groups of compound sharing similar physicochemical characteristics (Houghton, P. & Raman, A., 1998). This process is called fractionation and can be performed in various ways. For crude oil, there are few common ways that are done for laboratory studies. Fractionation is needed in this project to perform the Gas Chromatography-Mass Spectrometry (GCMS).

2.1.1 High Performance Liquid Chromatography

High Performance Liquid Chromatography (HPLC) is a popular method of analysis because it is easy to use and is not limited by the volatility or stability of the sample compound (High Performance Liquid Chromatography (HPLC): A Users Guide). High performance liquid chromatography is basically a highly improved form of column chromatography. Instead of a solvent being allowed to drip through a column under gravity, it is forced through under high pressures of up to 400 atmospheres. That makes it much faster. It also allows you to use a very much smaller particle size for the column packing material which gives a much greater surface area for interactions between the stationary phase and the molecules flowing past it. This allows a much better separation of the components of the mixture (HIGH PERFORMANCE LIQUID CHROMATOGRAPHY – HPLC).

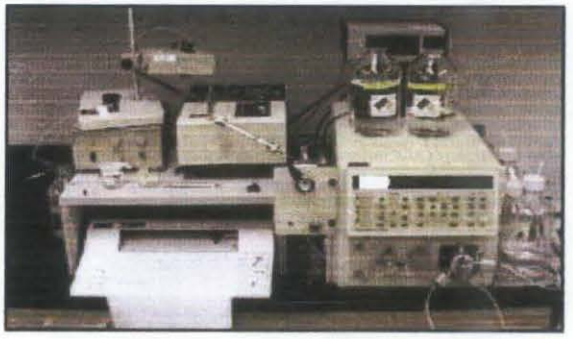

Figure 2.1: A HPLC equipment.

For this project, the crude oil is believed to contain high end of hydrocarbons which range starts from C_{20+}. The current standard that is available in UTP only runs for light hydrocarbon until C_{23}. A new standard needs to be purchased to use the same instrument.

2.1.2 Thin Layer Chromatography

Thin Layer Chromatography (TLC) is a simple, quick, and inexpensive procedure that gives the chemist a quick answer as to how many components are in a mixture (CUBoulder Organic Chemistry Undergraduate Courses). TLC is also used to support the identity of a compound in a mixture when the R_f (retention factor) of a compound is compared with the R_f of a known compound which preferably, both are run on the same TLC plate.

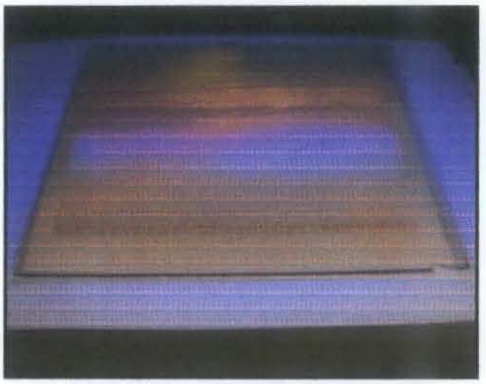

Figure 2.2: A TLC equipment.

A TLC plate is a sheet of glass, metal or plastic which is coated with a thin layer of a solid adsorbent (usually silica or alumina). A small amount of the mixture to be analyzed is spotted near the bottom of this plate. The TLC plate is then placed in a shallow pool of a solvent in a developing chamber so that only the very bottom of the plate is in the liquid. This liquid, or the eluent, is the mobile phase, and it slowly rises up the TLC plate by capillary action.

As the solvent moves past the spot that was applied, equilibrium is established for each component of the mixture between the molecules of that component which are adsorbed on the solid and the molecules which are in solution. In principle, the components will differ in solubility and in the strength of their adsorption to the

adsorbent and some components will be carried farther up the plate than others. When the solvent has reached the top of the plate, the plate is removed from the developing chamber, dried, and the separated components of the mixture are visualized. If the compounds are coloured, visualization is straightforward. Usually the compounds are not coloured, so a UV lamp is used to visualize the plates. The plate itself contains a fluor which fluoresces everywhere except where an organic compound is on the plate.

For this project, the TLC will be run to fractionate the crude samples into aliphatic, aromatic and NSO groups to compare the bulk properties among the samples. After the TLC is done, the aliphatic group will be the samples that are going to be used in GCMS for biomarker study.

2.1.3 Soxhlet Extraction

Zakaria et al. (2000) described a method of fractioning crude oil developed by Tokyo University of Agriculture and Technology. It basically applies the traditional Soxhlet extraction with dichloromethane (DCM). The column used to perform the fractionation contains silica gel. The silica gel is essentially supposed to eliminate the most polar compounds. Hartmann et al. (2000) further described the method.

The method is practiced in mini scale laboratory study to evaluate the source rocks and their depositional environments of crude oils (Brookes, S., 2007). Carbon isotope analysis of the saturate and aromatic fractions of the oil as part of the testing process is used to identify if the oil is of terrestrial or marine origin. The carbon isotope analysis requires less than a milligram of each fraction for analysis so the two fractions are prepared by this method.

This involves using a glass pasteur pipette as column to hold a bed of activated silica gel. A few microlitres of the crude oil sample are placed on top of the bed and the saturate fraction eluted with hexane (Figure 2.3). Once all the saturates have been washed through, the aromatics are eluted with toluene (Figure 2.4). Each fraction is then blown dry under nitrogen and are then analysed.

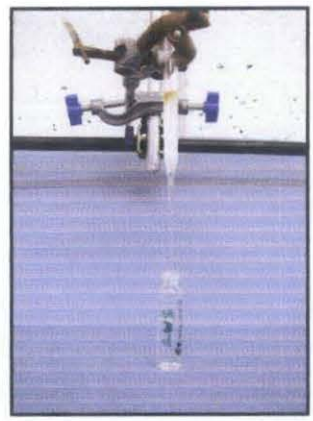

Figure 2.3: Saturate fraction of crude eluted with hexane.

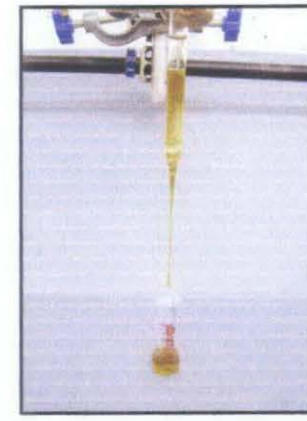

Figure 2.4: Aromatic fraction of crude eluted with toluene.

2.2 GAS CHROMATOGRAPHY METHOD

The most effective way to trace oil source for the past decade has always been gas chromatography (GC). GC is a method of separation which employs a gas mobile phase and a liquid adsorbed on a solid as a stationary phase (Chromatography-online). It is capable of separating very complex mixtures and the selectivity can be adjusted to separate almost any given pair of solutes by sensible choice of the stationary phase.

Figure 2.5: A gas chromatograph.

The process of gas chromatography is carried out in a specially designed instrument. A very small amount of liquid mixture is injected into the instrument and is volatilized in a hot injection chamber. Then, it is swept by a stream of inert carrier gas through a heated column which contains the stationary, high-boiling liquid. As the mixture travels through this column, its components go back and forth at different rates between the gas phase and dissolution in the high-boiling liquid, and

thus separate into pure components. Just before each compound exits the instrument, it passes through a detector. When the detector "sees" a compound, it sends an electronic message to the recorder, which responds by printing a peak on a piece of paper (CUBoulder Organic Chemistry Undergraduate Courses).

Gas chromatography mass spectrometry (GCMS) is an instrumental technique, comprising a gas chromatograph (GC) coupled to a mass spectrometer (MS), by which complex mixtures of chemicals may be separated, identified and quantified (GC/MS). This makes it ideal for the analysis of the hundreds of relatively low molecular weight compounds found in environmental materials.

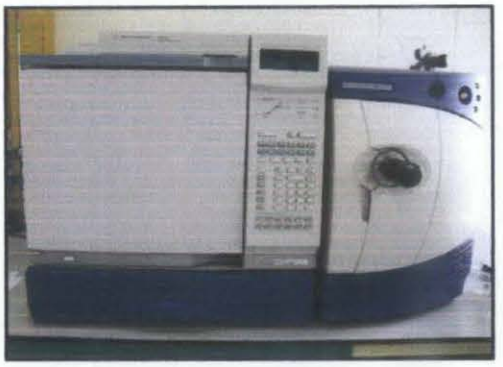

Figure 2.6: A GCMS equipment.

In order for a compound to be analysed by GCMS it must be sufficiently volatile and thermally stable. Besides, functionalised compounds may require chemical modification, prior to analysis, to eliminate undesirable adsorption effects that would otherwise affect the quality of the data obtained. Samples are usually analyzed as organic solutions consequently materials of interest (e.g. soils, sediments, tissues etc.) need to be solvent extracted and the extract subjected to various 'wet chemical' techniques before GCMS analysis is possible.

The sample solution is injected into the GC inlet where it is vaporized and swept onto a chromatographic column by the carrier gas. The sample flows through the column and the compounds comprising the mixture of interest are separated by virtue of their relative interaction with the coating of the column and the carrier. The latter part of the column passes through a heated transfer line and ends at the entrance to ion source where compounds eluting from the column are converted to ions.

The mass analyser (filter) separates the positively charged ions according to various mass related properties depending upon the analyser used. The most analysers common are quadrupoles and ion traps. After the ions are separated they enter a detector the output from which is amplified to boost the signal. The detector sends information to a computer that records all of the data produced, converts the electrical impulses into visual displays and hard copy displays. In addition, the computer also controls the operation of the mass spectrometer.

Below are the tables that show the bulk and molecular properties of oil samples from Semarang, Sumandak and Kinabalu fields. (Hannis, E., 2009)

Table 2.1: Bulk properties of the three fields' oil samples.

Location	Well	Aliphatic (%)	Aromatic (%)	NSO (%)	Ratio of Aliphatic To Aromatic	Oil sample description
East Baram Delta	Semarang Kecil 3A	68.83	24.68	6.49	2.2 : 1	Brown Orange, Liquid at room temperature
	Sumandak -1	57.23	35.54	7.23	1.6 : 1	Dark Brown, Liquid at room temperature
	Sumandak Tepi-1	57.33	31.33	11.33	1.8 : 1	Dark brown, Liquid at room temperature
	Kinabalu 1 (KNB 1)	52.99	34.33	12.69	1.5 : 1	Dark brownish black, Liquid at room temperature

Table 2.2: Molecular properties of the three fields' oil samples.

Location	Well	Pr/Ph	Pr/nC17	Ph/nC18	CPI
East Baram Delta	Semarang Kecil 3A	4.50	1.00	0.20	1.26
	Sumandak-1	BIODEGRADED			
	Sumandak Tepi-1	BIODEGRADED			
	Kinabalu 1 (KNB 1)	4.60	6.20	1.60	1.07

CHAPTER 3
METHODOLOGY

3.1 RESEARCH METHODOLOGY

The background of the project is studied thoroughly before coming up with a clear definite problem statement. Then, objectives of the project are identified to define the purpose of the research. Literature review is done next to study more about the current material that describes the project. Hence, methodology is structured to provide a comprehensible guideline on how to conduct the research and fulfil the objectives. The results are documented and discussed in detail to yield an acceptable conclusion and recommendations in order to complete the project. Finally, a report is produced to compile all the literature review and research findings for future reference.

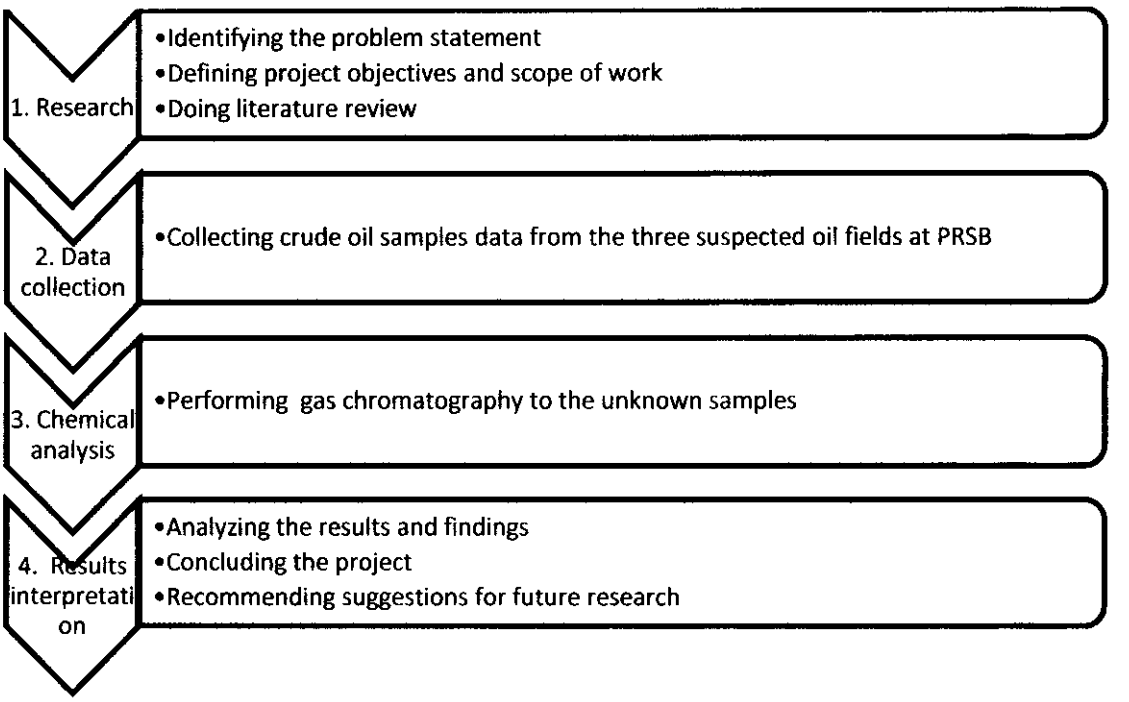

Figure 3.1: A brief research methodology.

3.2 PROJECT ACTIVITIES

Current GC and GCMS data of source oil from three oil fields adjacent to Labuan which are Sumandak, Semarang and Kinabalu are collected from PETRONAS Research and Services Sdn. Bhd., Bangi; and so do other crude samples from Angsi, Baronia, Dulang, Bokor and Tapis. Next, all tools, equipment and chemicals that are needed in the analysis are ordered before the analysis started. After that, the unknown samples must be extracted and fractioned. Then only chemical analysis can be performed unto all the oil samples. The results of the three crude samples are compared to the ones of the three unknown samples.

3.2.1 Extraction of Oil Samples

1. The unknown oil samples were labelled **A**, **B**, **C**. Each of the samples was precisely weighted at 10 mg.
2. A pipette column was filled with 5% H_2O deactivated silica gel.
3. **A** was dissolved in 1 mL of DCM/hexane (1:3, v/v).
4. **A** was pipetted onto the top of the column.
5. The stopcock was opened and then closed to allow **A** to penetrate the silica gel surface.
6. Hydrocarbons ranging from n-alkanes to polycyclic aromatic hydrocarbons (PAHs) were eluted with 20 mL of DCM/hexane (1:3, v/v) into a 50 mL round bottom flask.
7. The sample was reduced in volume to ~ 5 mL and rotoevaporated just to dryness by a rotary evaporator to remove the solvent.
8. Step 3 until 8 were repeated for the rest of the oil samples.
9. All extracted oil samples were stored in a 10 mL glass vial each.

Figure 3.2: Extracted oil samples.

3.2.2 Gas Chromatography of Oil Samples

After the extraction, the samples were all ready to run the gas chromatography. The normal alkanes of the oil seep samples were analyzed on Hewlett Packard series II 5890 gas chromatograph with helium as the carrier gas at 200 kPa. The injection port was maintained at 350 °C and the 10 µl samples were injected with splitless mode followed by 1 min purge after injection. Column temperature was held at 30 °C for 1 min, then programmed at 4 °C/min to 320 °C and held for 110 min. The detector temperature was held at 350 °C. Normal alkanes ranging from C_8 to C_{35}, pristane and phytane were determined. Identification of sample peaks was based on comparison of retention time of the authentic standard run on the same day.

3.3 KEY MILESTONES

There are a few key milestones throughout this project. Progress report I should be submitted in the fifth week of the semester. Progress report II on the other hand should be turned in the eleventh week of the semester. Final report finally must be submitted in the fourteenth week followed by oral presentation on the eighteenth week. There are also going to be poster exhibition held for presentation of the progress of the project. (APPENDIX)

3.4 TOOLS AND EQUIPMENT

Table 3.1: The tools, equipment and chemicals required for the chemical analysis.

	Equipment	Tools	Chemicals
Extraction	Balance. Rotary evaporator.	Pasteur pipettes. Beakers. Measurement cylinders. Crimpers. Pipettes. Round bottom flasks. Dark glass bottles.	5% H_20 deactivated silica gel. Dichloromethane (DCM). Hexane.
GC	HP series II 5890	Syringes. Glass vials.	Helium. DCM. Toluene.

CHAPTER 4
RESULTS AND DISCUSSION

The gas chromatograms for the three unknown crude samples and the eight oil fields crude samples are shown below. From all the chromatograms, we can see the highest peek at minute 7 is the solvent (DCM) peak. Pristane starts to emerge after C17 while phytane after C18 at around minute 55. Sample C is waxy therefore the peaks that are dominant starts at C12 while sample A and sample B the peaks starts at C8. Besides, toluene is eluted after DCM at minute 15.

The chromatogram of Sumandak sample clearly shows that the crude is biodegraded as the peaks are very erratic; there is presence of hump and decreasing of n-alkanes members. Hence, it can be concluded that the three unknown samples are not from Sumandak field. From the chromatogram of Kinabalu sample, the pristane peak is very dominant than C17. Thus, early conclusion can be drawn that the three unknown samples are not from Kinabalu field.

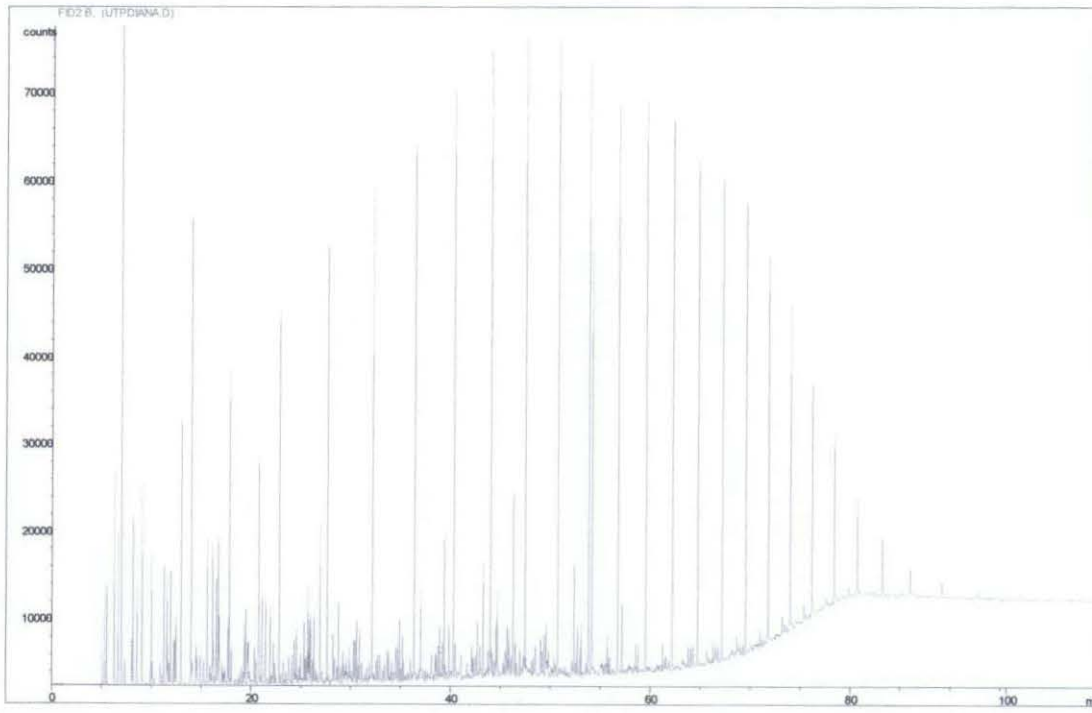

Figure 4.1: Gas chromatogram of sample A.

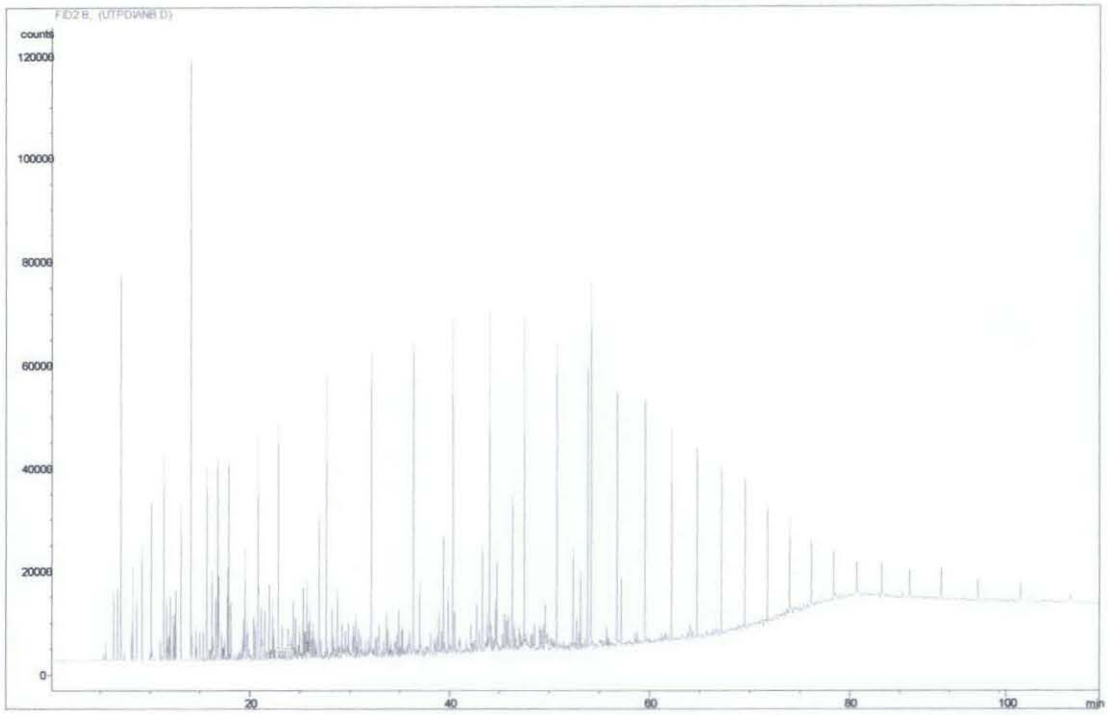

Figure 4.2: Gas chromatogram of sample B.

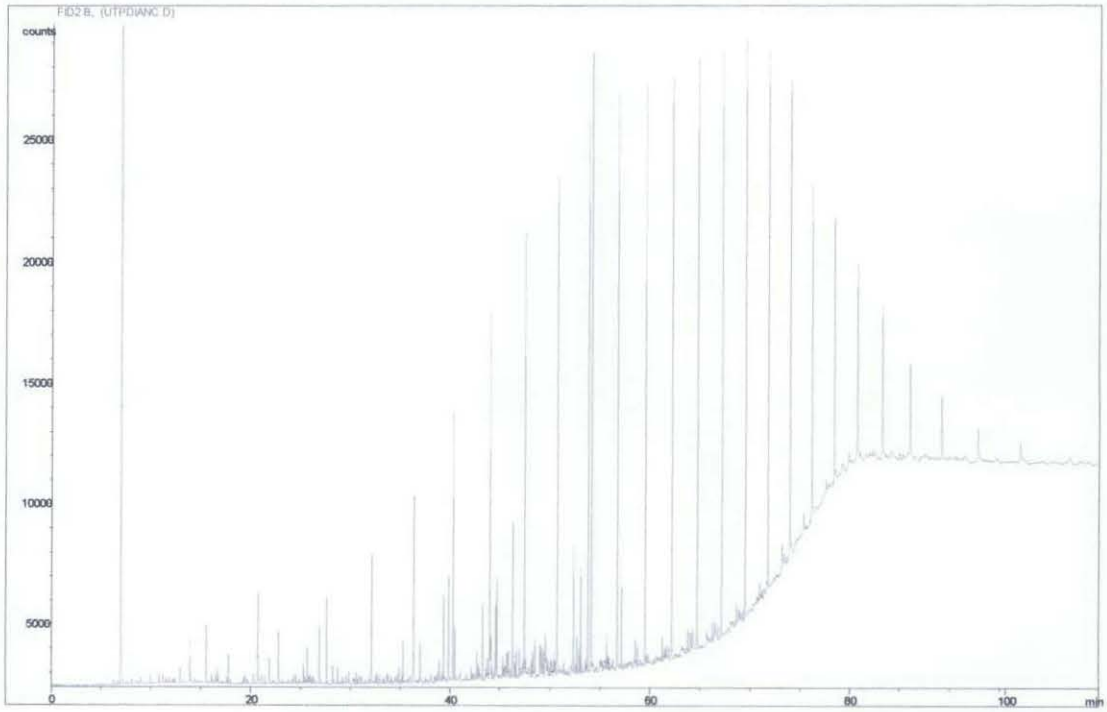

Figure 4.3: Gas chromatogram of sample C.

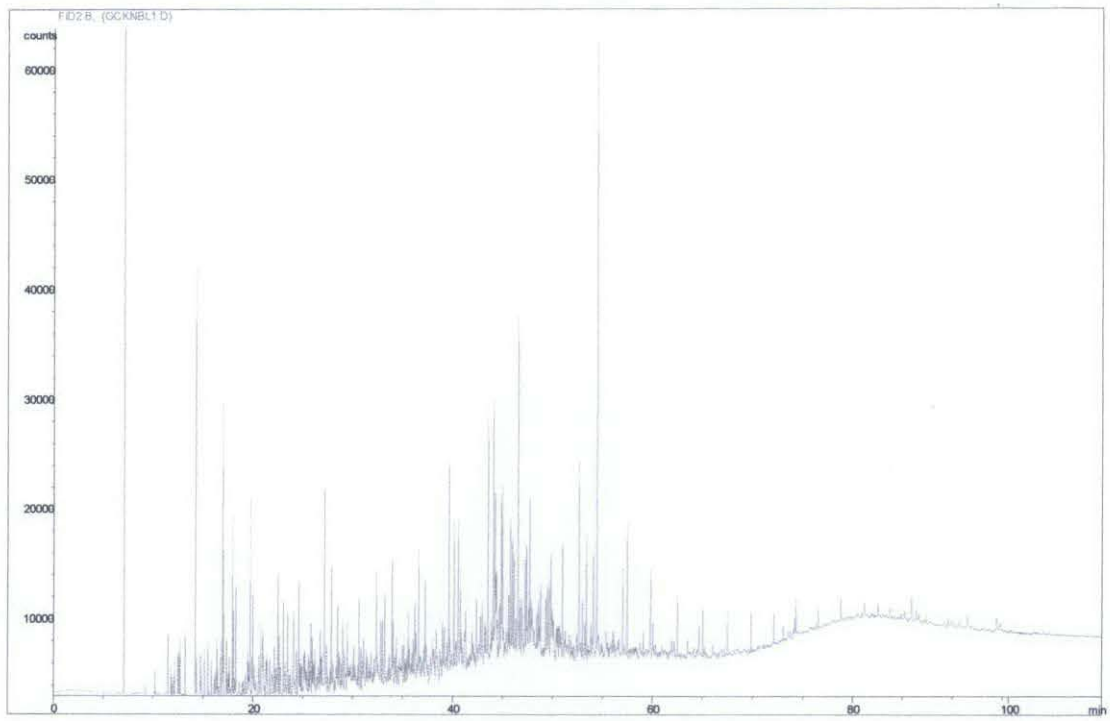

Figure 4.4: Gas chromatogram of sample Kinabalu.

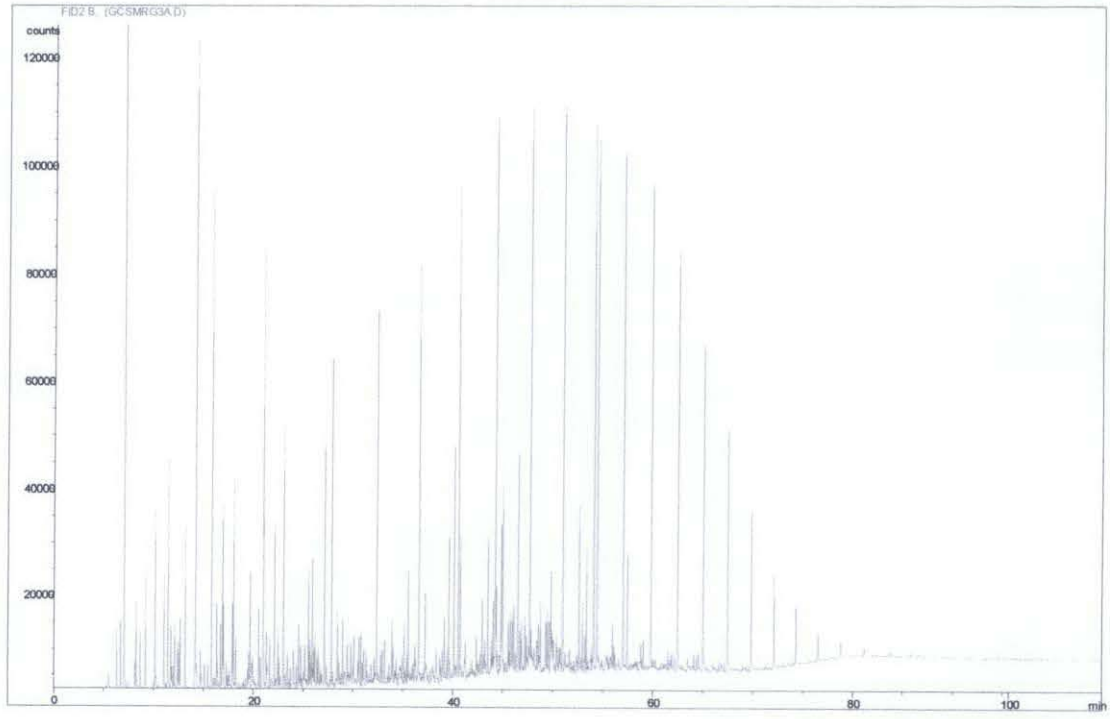

Figure 4.5: Gas chromatogram of sample Semarang.

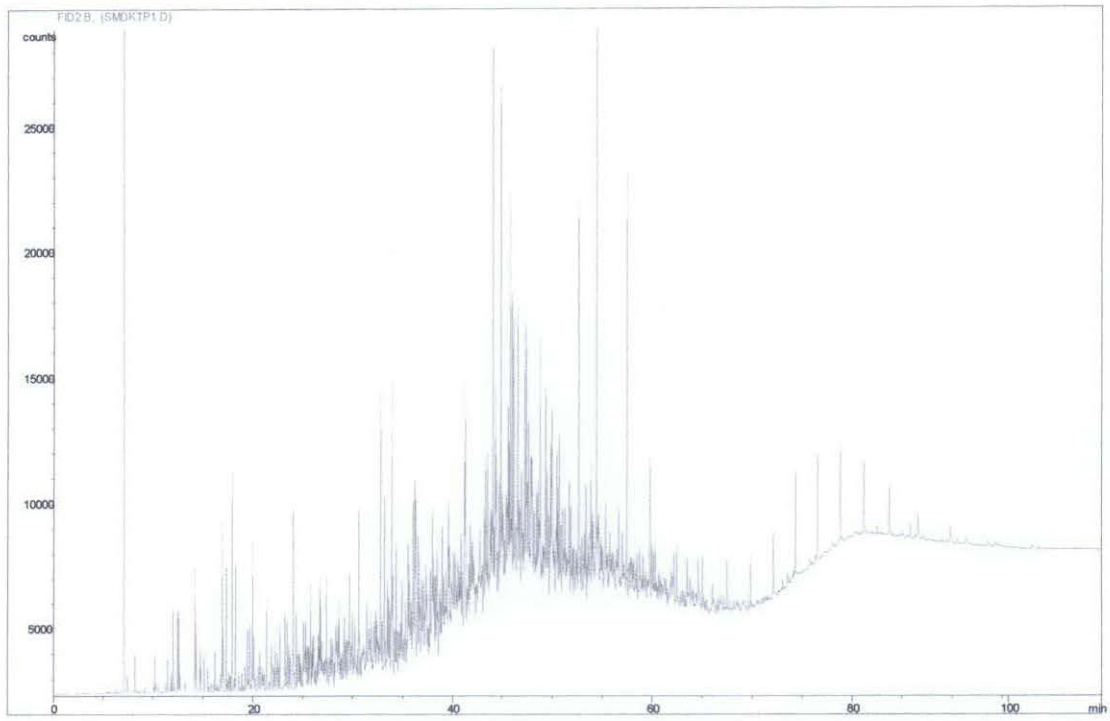

Figure 4.6: Gas chromatogram of sample Sumandak.

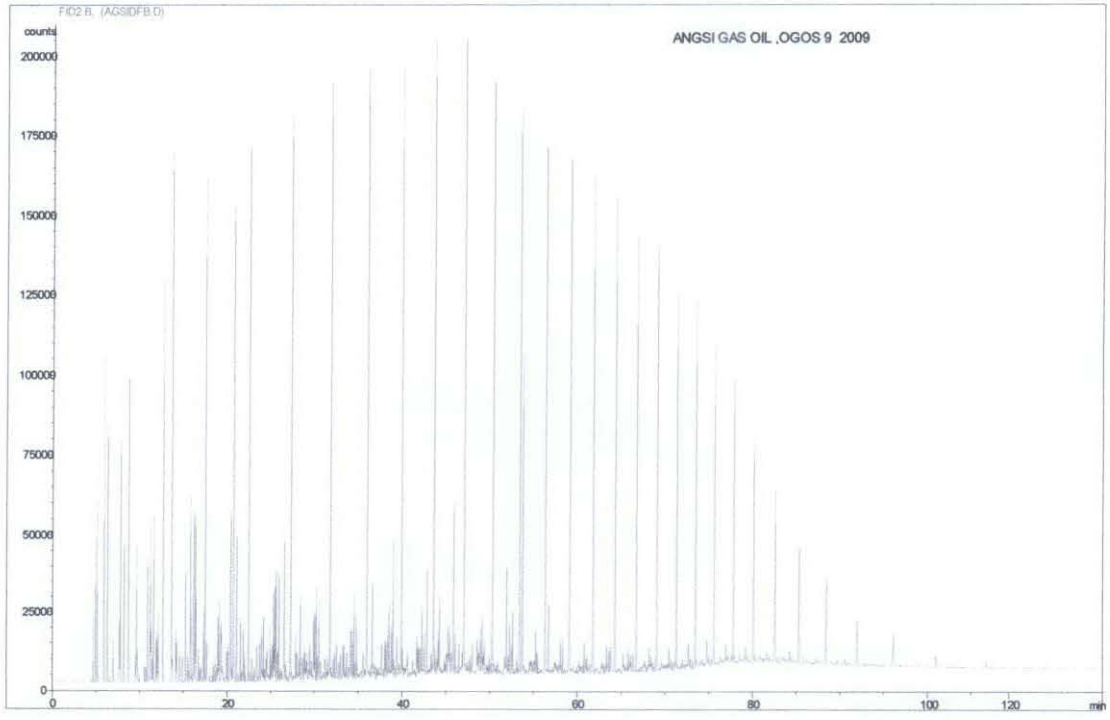

Figure 4.7: Gas chromatogram of sample Angsi.

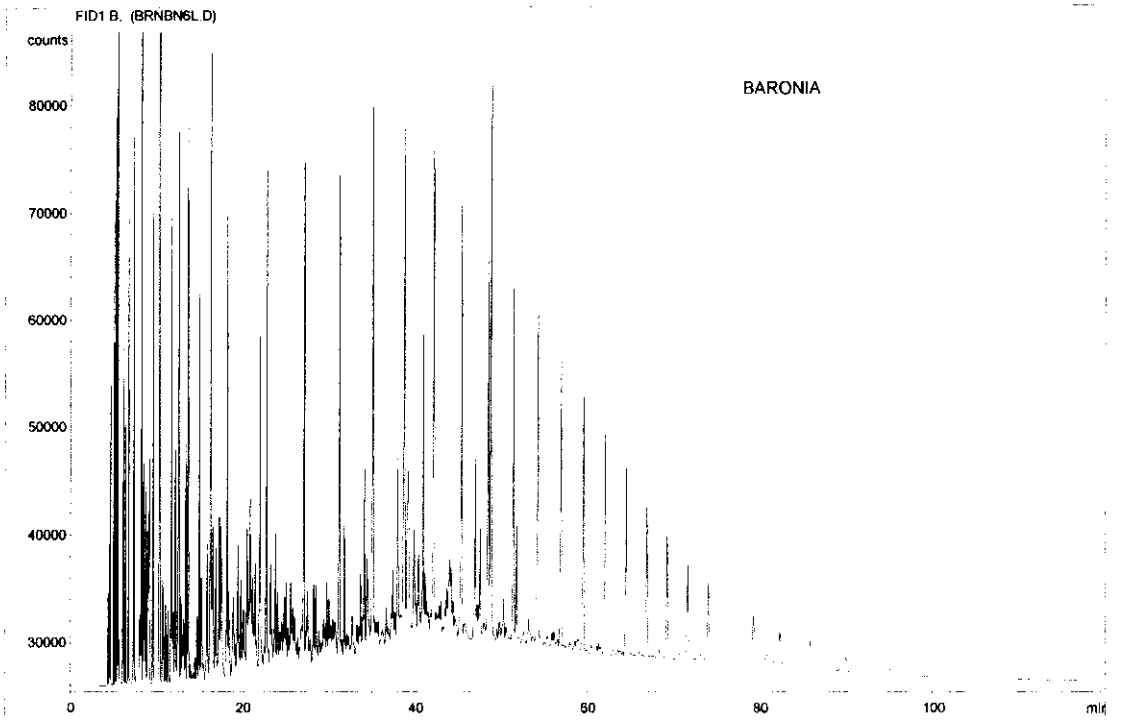

Figure 4.8: Gas chromatogram of sample Baronia.

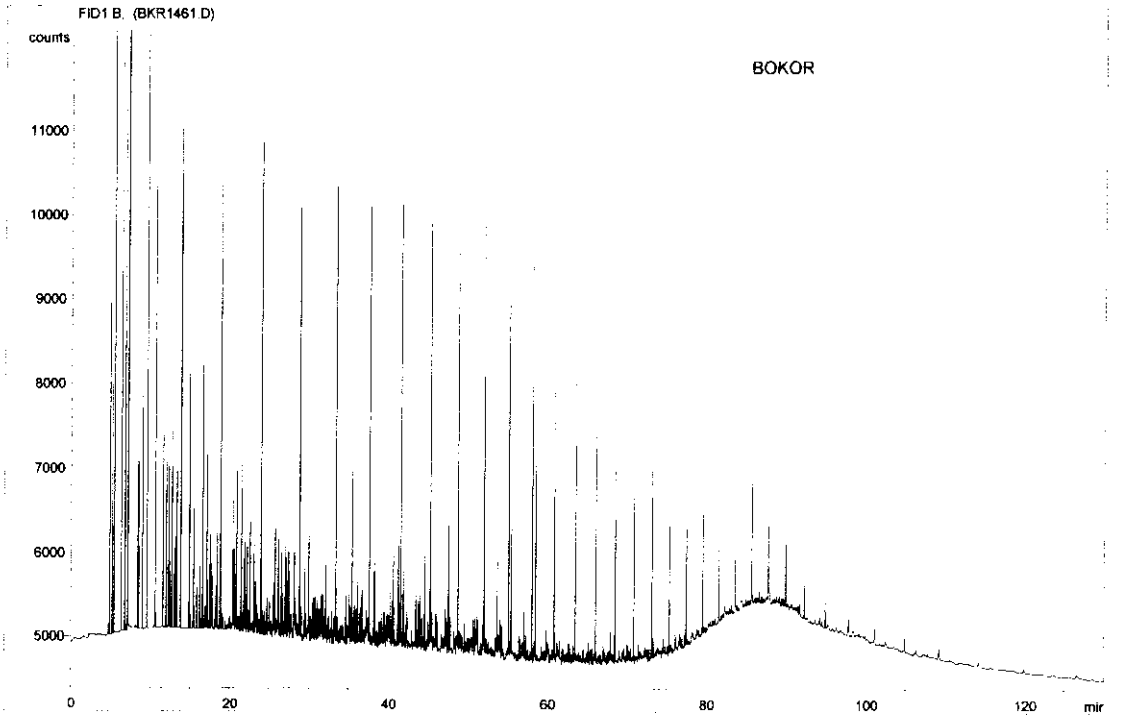

Figure 4.9: Gas chromatogram of sample Bokor.

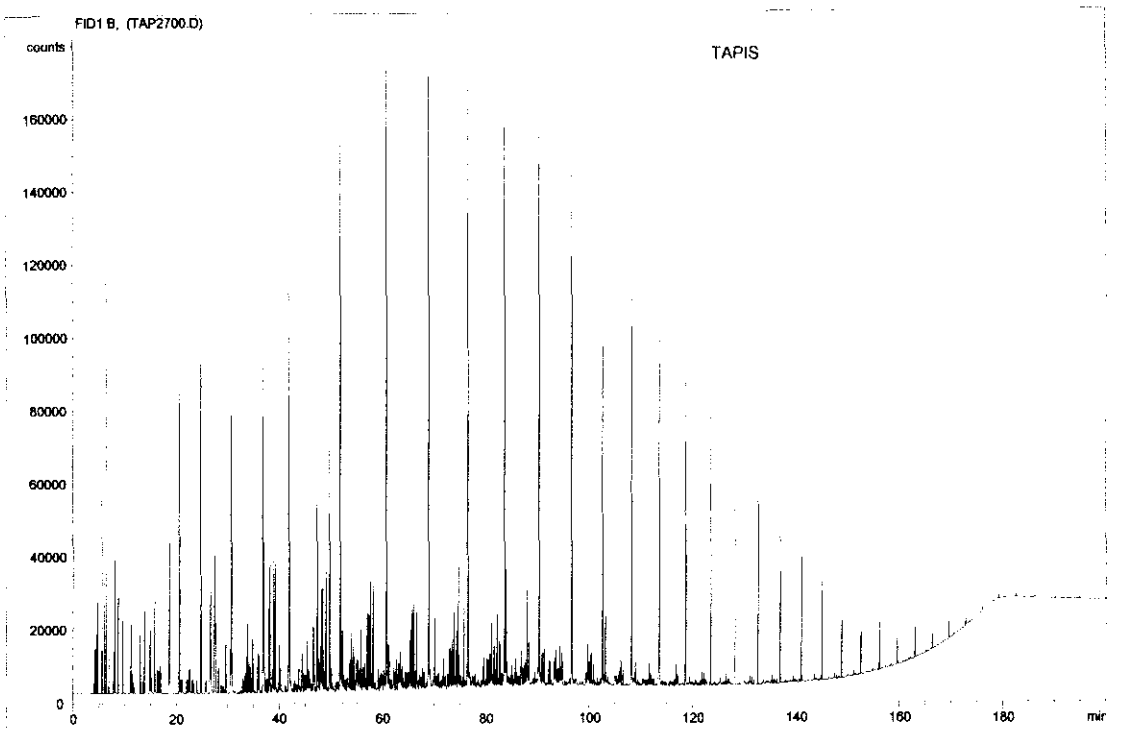

Figure 4.10: Gas chromatogram of sample Tapis.

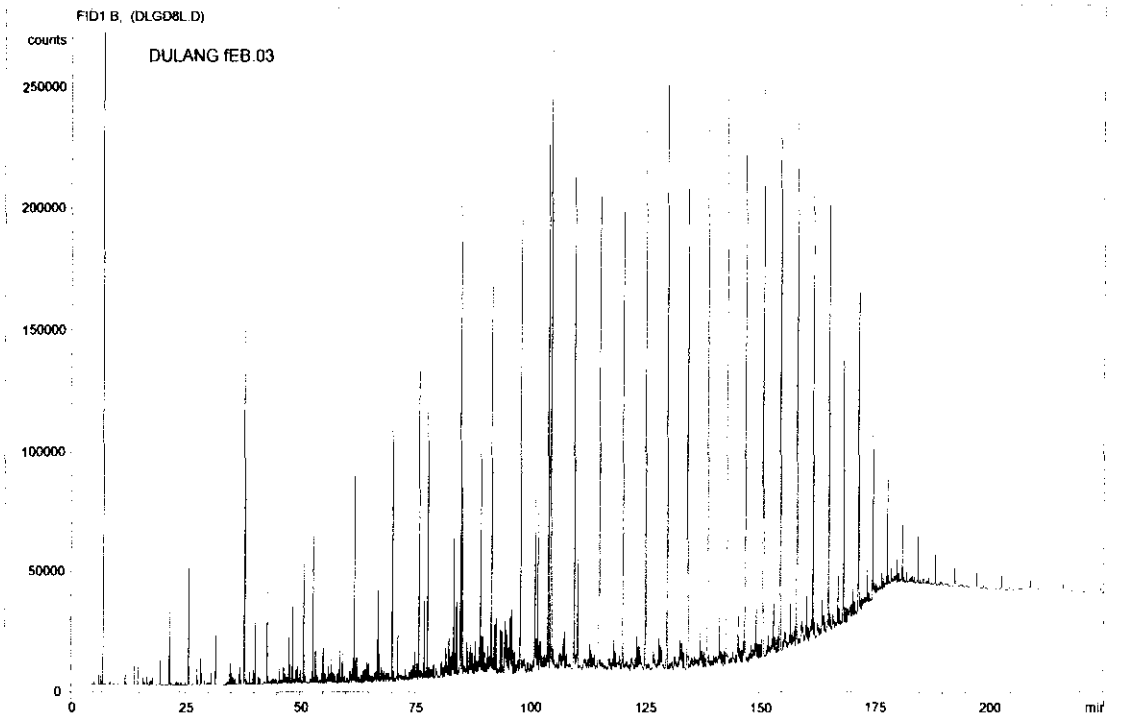

Figure 4.11: Gas chromatogram of sample Dulang.

Below is the table that shows result from the GC run for the three unknown samples. Values for Kinabalu, Semarang and Sumandak are taken from Table 2.2. The L/H values for them are calculated and added to the table below. Additional GC results of crude samples from Angsi, Baronia, Dulang, Bokor and Tapis are also included.

Table 4.1: Molecular properties of the crude samples.

Sample	Pr/Ph	Pr/C17	Ph/C18	CPI	L/H
A	6.00	0.70	0.12	1.18	8.39
B	5.47	1.28	0.26	1.17	7.06
C	7.27	1.09	0.14	1.16	4.91
KINABALU	4.6	6.2	1.60	1.07	3.50
SEMARANG	4.5	1	0.20	1.26	2.94
ANGSI	5.65	0.56	0.13	1.10	3.64
BARONIA	5.00	1.31	0.32	1.11	7.78
DULANG	6.28	1.11	0.11	1.09	1.43
BOKOR	0.61	0.33	0.54	0.88	3.39
TAPIS	1.56	0.17	0.15	1.03	6.53
SUMANDAK	Biodegraded				

*CPI = ½[(C25+C27+C29+C31+C33)/(C24+C26+C28+C30+C32) + (C25+C27+C29+C31+C33)/(C26+C28+C30+C32+C34)]
**L/H = (∑ C16 to C25) / (∑ C26 to C35)

All of the samples with exception of Sumandak has high Pr/Ph ratio between 2.5 to 7.0. These indicate that they all come from oxidizing terrestrial environment. However, Tapis and Bokor have lower ratio. This shows that the A, B and C samples do not come from Tapis and Bokor fields. Carbon Preference Index (CPI) value more than 1.0 indicates a mature oil characteristic.

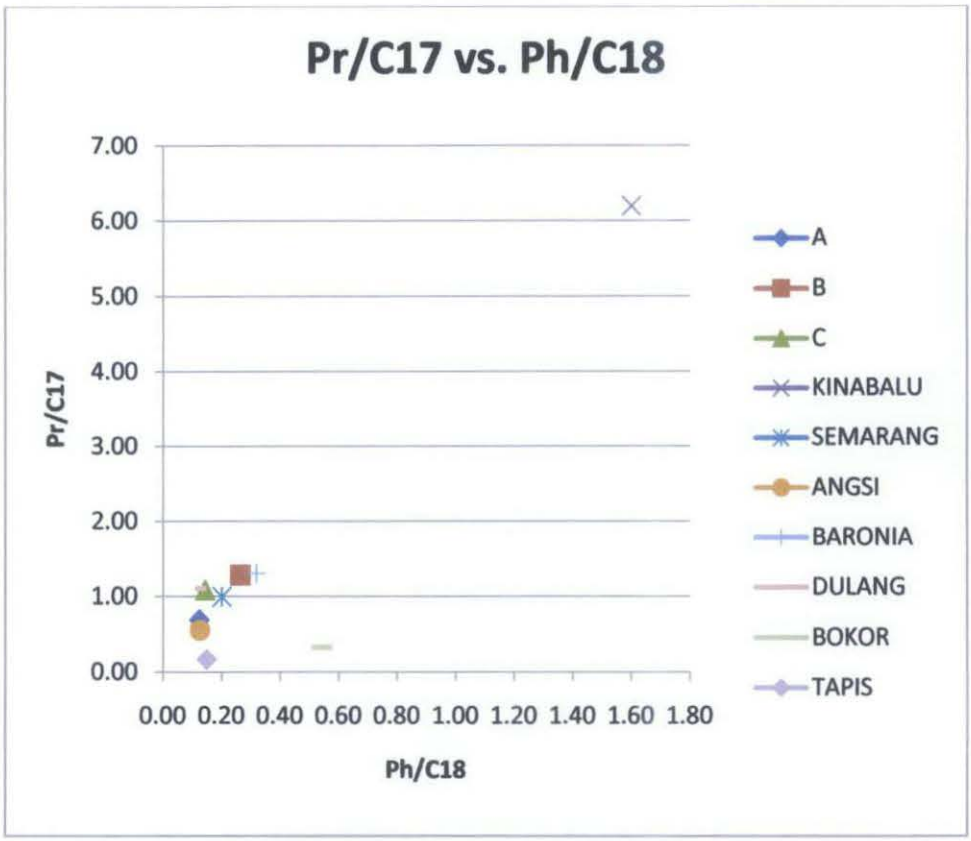

Figure 4.12: Pr/C17 against Ph/C18.

From Figure 4.12, the Kinabalu sample clearly shows that A, B and C do not belong to its field. The A sample falls into Angsi while the B sample overlaps with Baronia. The C sample on the other hand matches Dulang. Thus, it can be concluded, that sample A is from Angsi field, sample B is from Baronia field while sample C is from Dulang field.

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

It can be concluded that the source of the three unknown crude do not come from Sumandak field as the crude of Sumandak is biodegraded. From the study of the alkane relative distribution, it is also concluded that the three unknown crude do not belong to the rest of the two fields which are Kinabalu and Sumandak. When GC is performed onto the other probable samples, it is proven that the A sample is from Angsi field, the B sample is from Baronia while the C sample is from Dulang. These show that the three A, B and C samples do not come from the oil seep in Labuan.

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APPENDIX

Action Item	Action By	Date	Note
Briefing & update on students progress	Coordinator / Students / Supervisors	27-Jan-10	WEEK 1
Project work commences	Students	-	WEEK 2 -4
Submission of Progress Report 1	Students	25-Feb-10	WEEK 5
Submission of Progress Report 1 Marks	Supervisors / FYP Committee	5-Mar-10	WEEK 6
Submission of Progress Report 2 (Draft of Final Report)	Students	9-Apr-10	WEEK 11
Poster Exhibition / Pre-EDX/ Progress Reporting	Students / Coordinator	08 & 09 April 2010	WEEK 11
EDX	Selected Students/ Coordinator		WEEK 12
Submission of Final Report (CD Softcopy & Softbound)	Students / Supervisors	30-Apr-10	WEEK 14
Delivery of Final Report to External Examiner / Marking by External Examiner	FYP Committee / Coordinator	7-May-10	WEEK 15
Final Oral Presentation	Students/ Supervisors / Internal & External Examiners/ FYP Committee	24 May - 4 June 2010	WEEK 18 - 19
Submission of hardbound copies			